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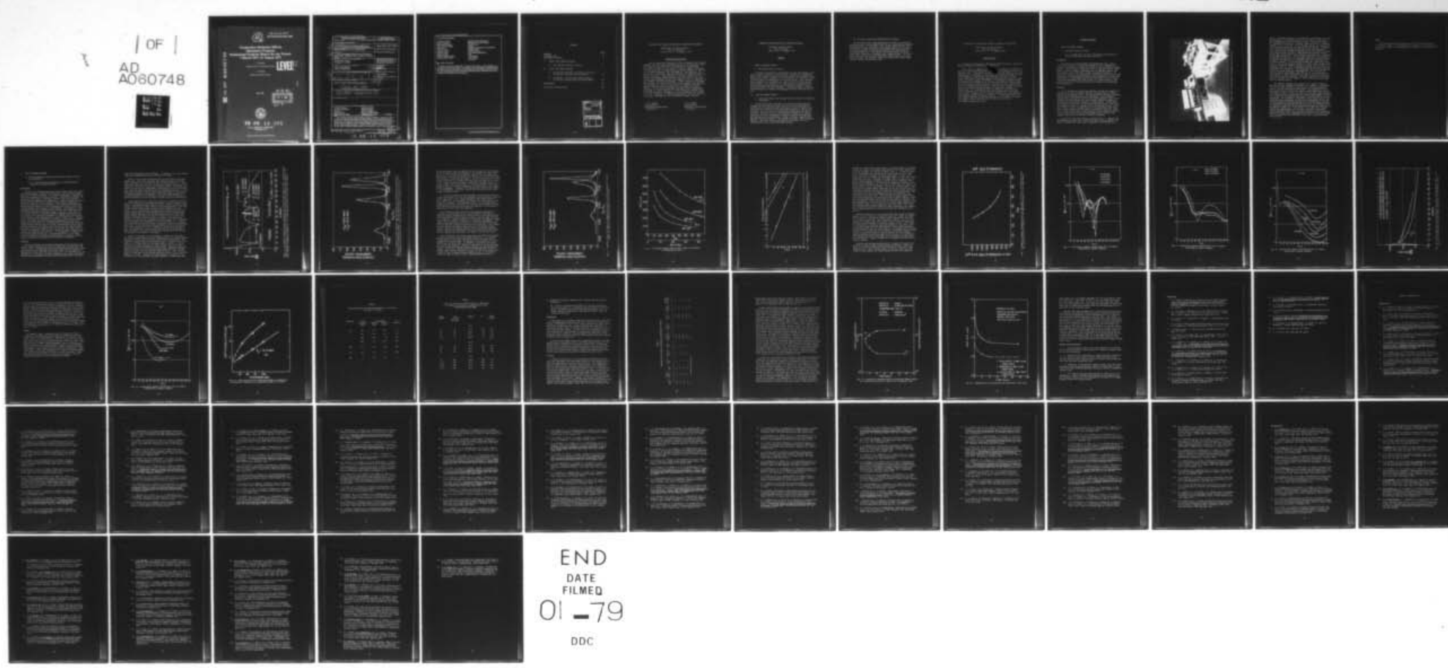

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NRL Memorandum Report 3806

Cooperative Radiation Effects Simulation Program Semiannual Progress Report for the Period: 1 March 1977–31 August 1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The Cooperation Radiation Effects Simulation Program (CORES) is a collaborative effort of the Engineering Materials and Radiation Technology Divisions of NRL Materials and General Sciences Area. The goal of the research is to provide the theoretical and experimental bases for understanding the mechanisms of nuclear radiation damage of metals, as well as a theoretical insight into energy deposition processes. In this the Van de Graaff and Cyclotron are used to simulate rapidly the radiation damage produced over long periods in reactor neutron environments.
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19. Key Words (Continued)

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Radiation-induced defects
Radiation-induced displacements
Simulation of neutron irradiations
Structural alloys
Swelling
Temperature dependence of void formation
Transport theory
Vacancies in metals
Van de Graaff bombardments
Voids
Void formation
Void nucleation
Void growth

20. Abstract (Continued)

Progress for the period 1 March 1977 - 31 August 1977, includes: (1) the completion of a semiautomatic system for the analysis of transmission electron micrographs, (2) profiling hydrogen and helium in parts per million concentration by elastic scattering techniques, and (3) the microstructural characterization of stress and ion-induced transient creep in cold-worked nickel and its comparisons with predictions of creep models.

CONTENT

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
for the Period:
1 March 1977 - 31 August 1977

PROGRAM DESCRIPTION

The Cooperative Radiation Effects Simulation Program (CORES) was initiated voluntarily by five Branches from the Engineering Materials and Radiation Technology Divisions of NRL on the basis of their common interests in the problems of simulating radiation damage in metals. The program promotes the exchange of information, discussion of problems, and the pursuit of collaborative research efforts. Semiannually a written report is prepared containing those portions of the work of the participating Branches which are judged to be of interest to the damage simulation problem. Since research findings which apply to the objectives of one sponsor may also be of interest to others, the overall progress related to damage simulation is included in the written report. Several of the participating Branches have independent programs on other aspects of the radiation damage problem; when results obtained in these programs are judged to be of interest to CORES participants they may also be included, informally, in the CORES program reviews.

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COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
for the period:
1 March 1977 - 31 August 1977

SUMMARY

I. HEAVY ION DAMAGE STUDIES

A. Micrograph Analysis System

To speed the analysis of transmission electron micrographs, both fully automatic systems and semi-automatic systems were considered. While fully automatic systems are used in optical metallography, they possess some serious drawbacks in the extraction of quantitative microstructural data in transmission electron microscopy. Since programming a computer for recognition of complex and overlapping objects is difficult, it was decided that a semi-automatic system with operator measurements and automatic data recording for computer input would be the most flexible compromise. The semi-automatic system has been developed and a number of advantages have been realized in its use.

II. LIGHT ION DAMAGE STUDIES

A. Profiling Hydrogen and Helium Isotopes by Elastic Scattering Techniques

Elastic scattering techniques to profile helium and hydrogen isotopes in host metal foils have been reviewed to understand all possibilities of improving depth resolution. Depth resolutions improve with the reduction of incident beam energy due to energy loss increases, but at a sacrifice of decreased thicknesses that may be profiled. The largest factor limiting depth resolution is energy spread of the beam. Variations of depth resolutions with materials and thickness are understandable in terms of energy straggling and multiple scattering effects. The minimum detectable limit is approximately 1 appm, with depth resolutions of micrometers for foil thicknesses of mils at cyclotron beam energies.

B. Transient Irradiation-Induced Creep of Nickel

The microstructural characterization of stress and ion-induced transient creep in cold worked nickel has been completed and compared with predictions of creep models. The results agree with the conclusion that a climb-controlled glide mechanism should occur primarily during transient irradiation creep and be more pronounced for cold worked materials. The network dislocation density achieved a value within eight hours consistent with annealed nickel. It is concluded that the creep strains observed at time periods up to eight hours represent true transient creep with longer time periods representing a nearly steady-state creep.

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
for the Period:
1 March 1977 - 31 August 1977

INTRODUCTION

L. A. Beach, Coordinator, Materials Modification & Analysis Branch, Radiation Technolo[illegible] Division.

This report summarizes s[illegible]al research accomplishments of the interdivisional c[illegible]tive program now in its seventh year. The goal of this [illegible]ram has been to use ion simulation techniques to increase the understanding of neutron damage of materials for advanced nuclear systems. Light and heavy ion bombardment techniques are used to represent neutron damage in order to accelerate research and to permit an evaluation of critical parameters which might not be feasible using nuclear reactors at test facilities. In this program the techniques applied have advanced along with our growing understanding of radiation damage of materials. To complement the ion damage simulation are other programs such as theoretical evaluation of atomic collisions, computation of energy deposition, and parallel experiments using nuclear reactors. The productivity of the CORES program is illustrated by a full list of technical papers and presentations in the last section.

Note: Manuscript submitted June 9, 1978.

RESEARCH PROGRESS

I. HEAVY ION DAMAGE STUDIES

A. Micrograph Analysis System

(J. A. Sprague and J. R. Reed, Thermostructural Materials Branch, Engineering Materials Division)

Background

The extraction of quantitative microstructural data, such as voids, dislocations and precipitate parameters from transmission electron micrographs is a major bottleneck in many irradiation damage studies. This problem is especially acute in charged particle experiments, in which integral techniques such as immersion density are not feasible, due to the small volume of irradiated material. To speed the acquisition of quantitative transmission electron microscopy (TEM) data, a semi-automatic system has been assembled to record size distributions, three-dimensional spatial distributions and electron diffraction information for direct input to a small computer. The system increases both the speed and the flexibility of photographic data acquisition.

Progress

In the evaluation of potential methods to speed the analysis of TEM data, both fully automatic systems, involving direct computer reading of photographic plates, and semi-automatic systems, requiring some operator interpretation, were considered. Fully automatic systems, which are widely used in optical metallography, have a considerable speed advantage, but the currently available commercial systems have some serious drawbacks for TEM studies. Specifically, the variation of contrast across many micrographs makes it difficult to program a computer to recognize and to accurately measure a variety of defects; also, recognition of overlapping objects and rejection of some common artifacts cannot be adequately achieved. Therefore, it was decided that a semi-automatic system combining measurement of defects by an operator and automatic data recording for computer input would be the most flexible compromise.

An overall view of the system is shown in Fig. 1. Defect size measurements are made with a Zeiss TGZ-3 particle size analyzer, in which a variable-size light spot is matched to some feature of a

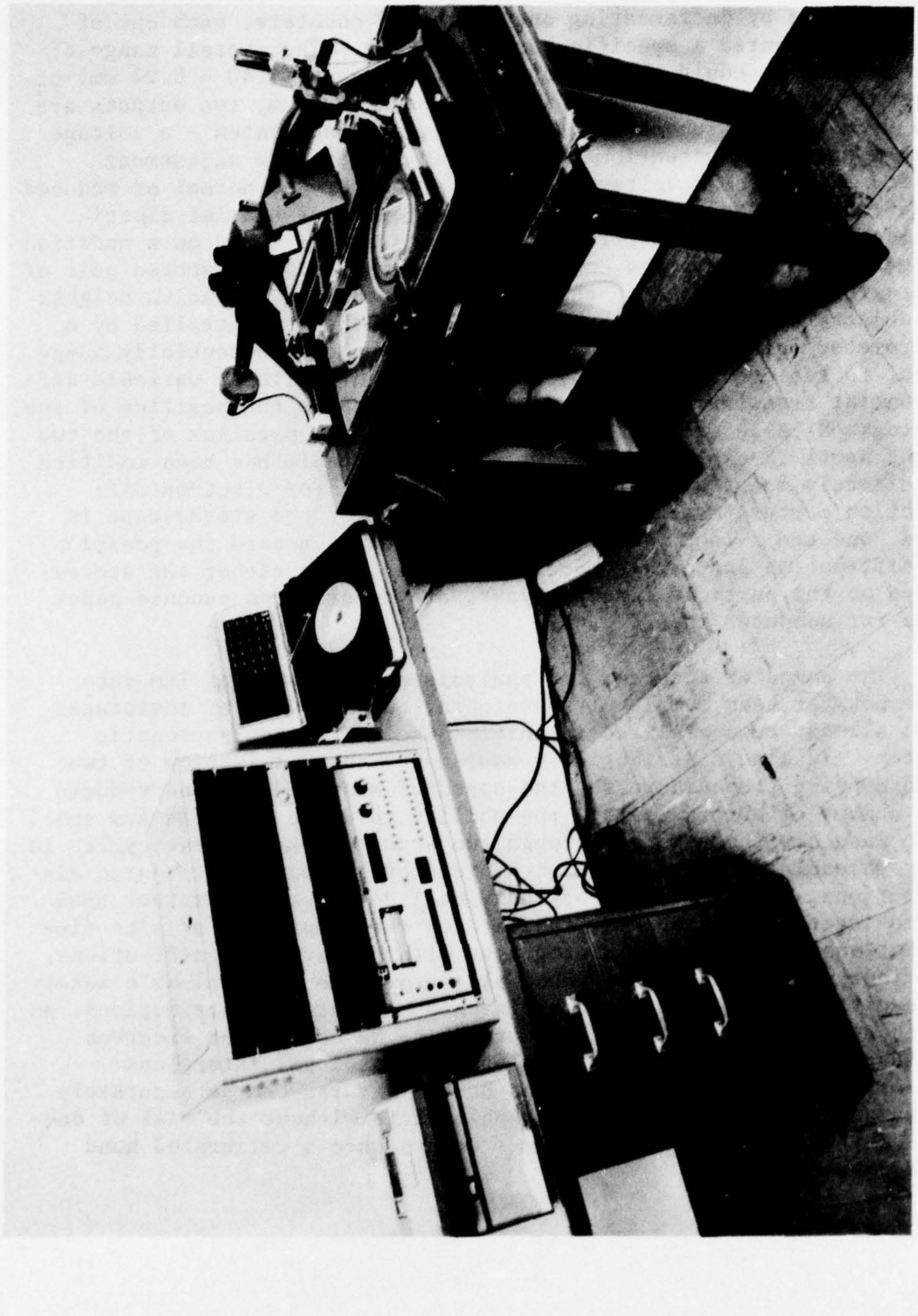

Fig. 1 - Overall view of semiautomatic micrograph analysis system

defect. In normal operation of the particle size analyzer, the size was recorded by incrementing one of the 48 counters, each one of which represented a specific range of sizes. The overall range of the instrument could be set to either "reduced" (0.40 - 9.24 mm) or "normal" (1.21 - 27.71 mm). In the present system, two outputs are fed from the instrument to the data acquisition system - a voltage from a precision potentiometer attached to the size adjustment shaft, and a second voltage indicating whether the normal or reduced size range has been selected. Three-dimensional spatial distributions and electron diffraction patterns are measured on a modified Hilger-Watts stereoscope. In its normal operation, a stereo pair of micrographs was placed on the pantograph table and relative heights of objects were measured by a floating light spot controlled by a micrometer spindle. In the modified stereoscope, essentially identical to the one described by Thomas (1), three linear variable differential transforms (LVDTs) are used to measure the position of the pantograph table (x- and y-coordinates) and the parallax of the two light spots (Z-coordinate). The pantograph table has been modified to directly mount glass-plate TEM negatives. For electron diffraction pattern measurement, only one side of the stereoscope is used, and the x- and y-coordinates are used to record the position of diffraction pattern features. Voltages, from either the stereoscope or the particle-size analyzer, are recorded on punched paper tape for computer input.

The computer programs for analysis of all types of TEM data have not yet been completely developed, but a number of advantages have already been realized in initial use of the semi-automatic system. In size distribution measurement, the elimination of two hand-copying steps increases the speed of measurements and reduces the number of errors. Also, the ability to use both large and small size ranges without going through two separate measurement cycles is very valuable on micrographs with broad or bi-modal defect size distributions. The recording of individual defect sizes, rather than fixed-increment histograms, facilitates the comparison of size distributions measured from micrographs with different magnifications. The instrumented stereoscope has also proven to be a valuable asset. In addition to measuring three-dimensional spatial distributions, as discussed by Thomas (1), it has proven to be useful for electron diffraction measurements, since lattice spacings, interplanar angles, specimen orientation, and other features can be accurately measured directly from a photographic plate without the risk of damage to the emulsion that sometimes occurs when a calibrated hand lens is used.

Plans

The micrograph analysis programs will be further developed as the system is used. If it appears to be desirable in the future, the system can be directly interfaced with a computer to provide on-line analysis.

II. LIGHT ION DAMAGE STUDIES

A. Profiling Hydrogen and Helium Isotopes by Elastic Scattering Techniques

(L. A. Beach, Materials Modification & Analysis Branch, Radiation Technology Division)

Background

Retention of hydrogen and helium in most metals and alloys causes deleterious changes in mechanical properties, such as embrittlement and loss of tensile strength. In a neutron environment, hydrogen and helium are generated by nuclear reactions. Interstitial hydrogen and helium are quite mobile in metallic lattices, and their mobility should increase smoothly with temperature. However, helium introduced into metals with sufficient energy to cause atomic displacement is known to accumulate or to be retained. The helium atoms become substitutional by trapping in lattice vacancies and these filled vacancies may migrate and coalesce to form bubbles. The mobility of this trapped helium does not significantly increase with temperature until the helium atoms have sufficient energy to pop out of the lattice. For fission reactors, including breeders, hydrogen generated by the (n,p) reaction is generally considered to diffuse from reactor materials because of its high mobility at reactor temperatures. Recently, it has been shown that hydrogen is retained at least at lower temperatures, in metals which have undergone predamage by heavier ions, particularly by helium (2). The generation of hydrogen and helium by 14-MeV neutrons will be greater in some thermonuclear devices than in breeders because of the higher cross sections at that neutron energy. The amount of hydrogen generated will be at least ten times that of helium. In addition, high fluxes of hydrogen isotopes, deuterium and tritium will bombard the first wall. It is important to understand how much hydrogen and helium is retained separately, or in combination at temperatures ranging from 500^{o}C down to cryogenic temperatures, and how this retention effects mechanical properties.

Progress

Profiling hydrogen and helium distributions in thin metallic samples by elastic scattering of light ion beams has been described previously (3-5). Hydrogen and helium concentrations greater than 1 appm can be profiled in reasonable accelerator times in foils limited in thicknesses to less than 10% of the range (6) of the incident ion. To determine the feasibility of higher resolutions by elastic scattering, several new measurements were made and much previous work, both pub-

lished and unpublished, were reviewed. In addition, it became obvious that some previously made points should be clarified.

Briefly, the incident beam penetrates the thin sample with small energy loss. Some of the incident ions undergo elastic scattering from a hydrogen or helium atom residing in the host material. After scattering, both the scattered ion and the recoiling struck atom lose energy at a higher rate than at the incident energy. The normal technique utilizes self-ion bombardment, scattering at 45^{o} in the laboratory, and coincident detection of the two outgoing particles; i.e., an alpha particle beam to profile helium concentrations or a proton beam to measure hydrogen concentrations. Since the summed energy of the two detected particles is related to the depth of the scattering, a depth distribution is derived from the total energy loss distribution.

Three figures of merit are often referred to: detection sensitivity, system resolution, and depth resolution. For the highest sensitivity, a bombarding energy should be selected where the elastic scattering cross section is high, and for clear interpretation, the cross section should remain nearly constant as the primary beam loses energy. For α-α scattering, the cross section at 90^{o} in the center-of-mass system exhibits rapid fluctuations at certain energies (6). Alpha energies available from the NRL cyclotron are from 25-75 MeV. Figure 2 shows more details of the α-α cross section at 90^{o} from 600 keV to 75 MeV as taken from the literature. The large oscillations in cross section correspond to excited states in the unstable ^{8}Be system which decay primarily into the two alphas, i.e., states with even spin and positive energy. The double resonance oscillation near 34 MeV is well documented by experimental points at intervals of 40 keV and corresponds to the 2^{+} doublet at 16.63 and 16.92 MeV in ^{8}Be. The variations in cross section near 40 MeV and 50 MeV may be more oscillatory than shown as phase shift analysis clearly indicate the multiple levels shown on the figure.

Pieper (4) did most of his profiling of samples near 49.0 MeV, but he also showed results of a 32.7-MeV profile of an aluminum sample implanted at front and back surfaces with 60-keV helium atoms. He noted the front peak was 44% of the back peak in contrast with a 85% ratio for a 49-MeV profile of the same sample - see Fig. 3. He attributed this large difference in front to back ratio to multiple scattering effects increasing at lower energies. However, some later unpublished work of his on several samples from ORNL with 30.3-MeV profiling clearly show front to back peak ratios of $\sim$ 85%. He also noted the data rate at 32.6 MeV was down by a factor of 2.7 from that observed at 49 MeV. Inspection of the parameters recorded for the 32.6-MeV run in the cyclotron log book indicate the beam energy most

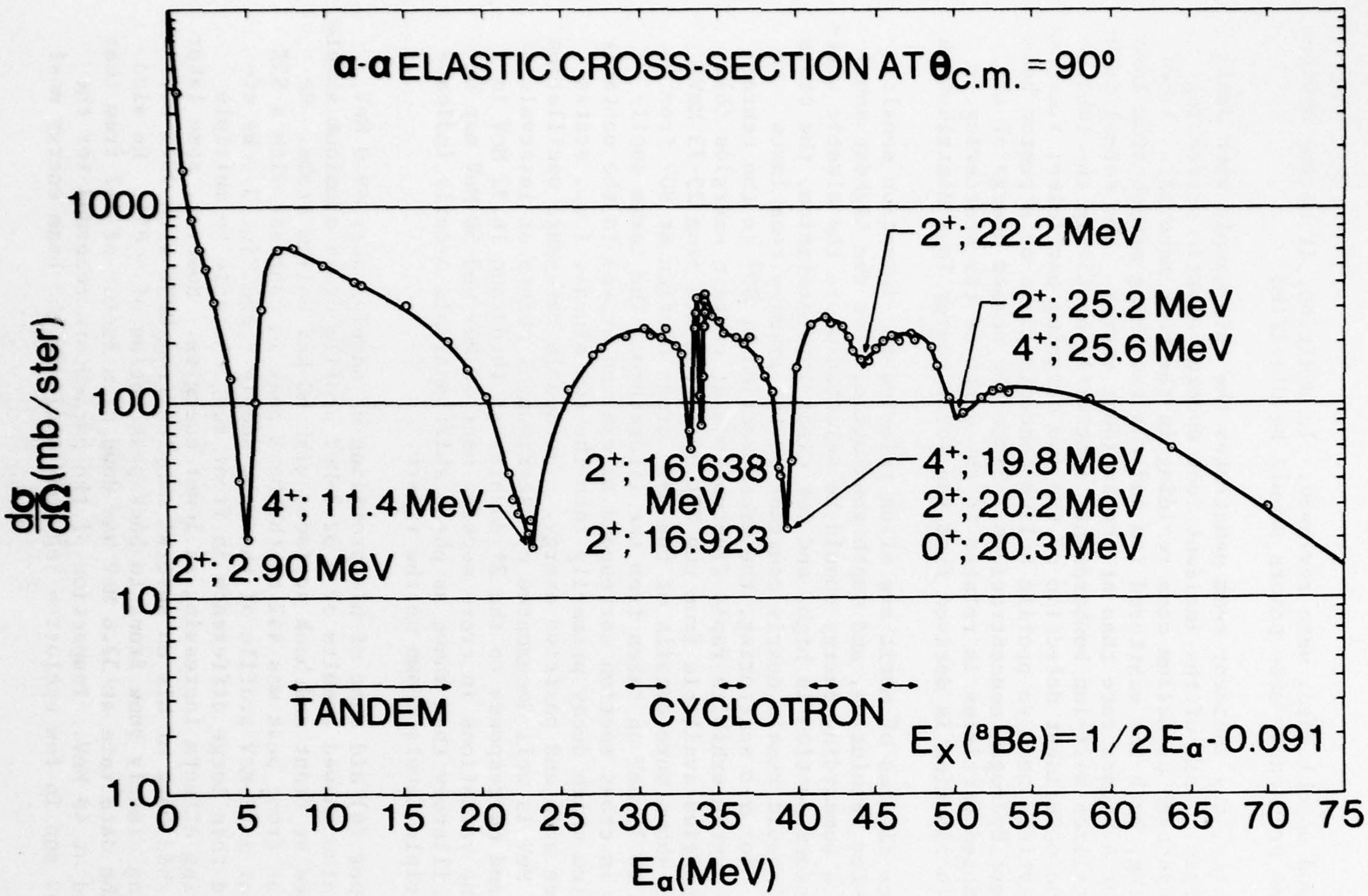

Fig. 2 - α-α elastic scattering cross section at the center-of-mass angle of 90°. The dips in the cross section are due to destructive interference of the even spin, positive parity states of ^{8}Be. Most desirable bombarding energies for α-α profiling are shown by the double ended arrows.

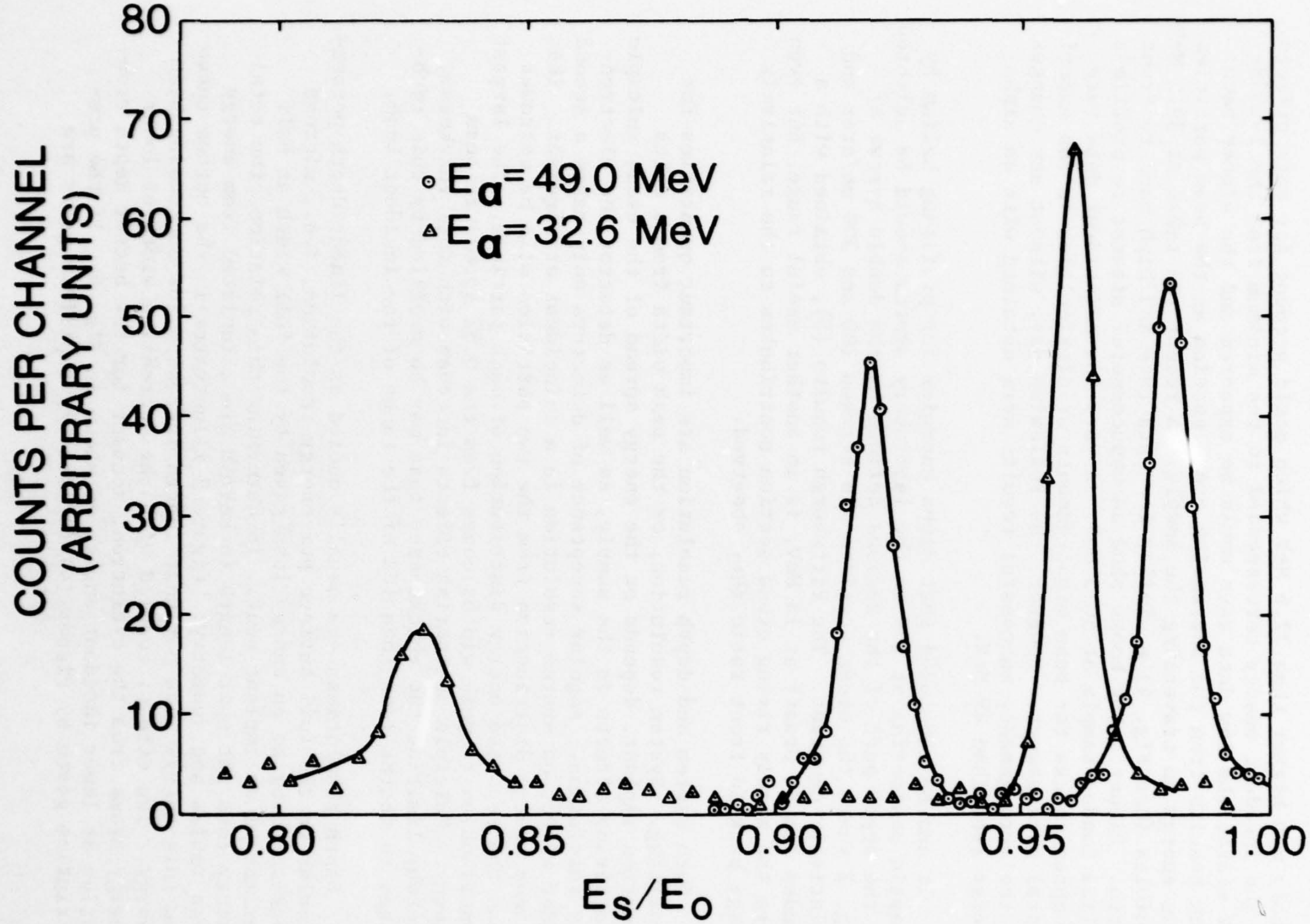

Fig. 3 - Profiles of helium implanted near the surface of an aluminum foil one-mil thick obtained at two bombarding energies. The poor front-to-back ratio at 32.6 MeV is due to an increasing cross section as the initial beam loses energy in traversing the foil.

likely was higher than 32.6 MeV which could account for both effects. If the incident energy corresponded to the minimum from the 16.638-MeV state, the low data rate would be expected and the higher back peak results from the increasing cross section as the beam particles lose energy in traversing the sample. A recent run taken at 39.3 MeV exhibits (see Fig. 4) a similar low data rate and high back to front ratio. Pieper's log books show an unsuccessful attempt to profile a helium loaded sample at 50.5 MeV. A very low coincident data rate prompted checks for beam misalignments or difficulties in the experimental arrangement. However, the following day, without any changes in the arrangement, successful results were obtained with an alpha energy less than 49 MeV.

It can be concluded that alpha energies for profiling helium by elastic scattering at 45° in the laboratory system should be selected in the upper part of the regions defined by the double arrows of Fig. 2 where the cross sections are between 200 and 300 mb/ster and relatively constant. The Pittsburgh results (5), obtained with a Tandem Van de Graaff at 18 MeV, is in another useful range, but even here the slowly rising cross section contributes to the relatively large back to front ratio they observed.

Both system and depth resolution are important quantities for profiling. System resolution, or the peak width from a delta function implant, depends on the energy spread of the beam, multiple scattering effects in the sample, as well as detector and electronics resolution. Angular acceptance of detectors only makes a second order effect on system resolution in a coincident arrangement. The summed energy distribution from the two particles will be narrower than the separate energy distribution of each particle. The largest contribution to peak widths comes from the 0.5% spread in beam energy. Multiple scattering effects increase with foil thickness, thereby limiting the thicknesses that may be profiled by this technique to depths less than 10% of the range of the incident beam.

Depth resolutions are usually quoted as the linear depth perpendicular to the foil surface per energy resolution, i.e., microns corresponding to an energy loss given by the full width at half maximum of an implant peak. To determine this relation, the total energy loss per unit length in keV/μM are calculated from energy loss tables and geometry. Figure 5 illustrates in the bottom curve how this energy loss per unit length increases for decreasing beam energy. The effect, coupled with the decreasing width of lower energy beams from the cyclotron, account for the better depth resolution at lower incident energy as shown in Fig. 6. If the prescription given by Pieper (4) for setting amplifier gains are

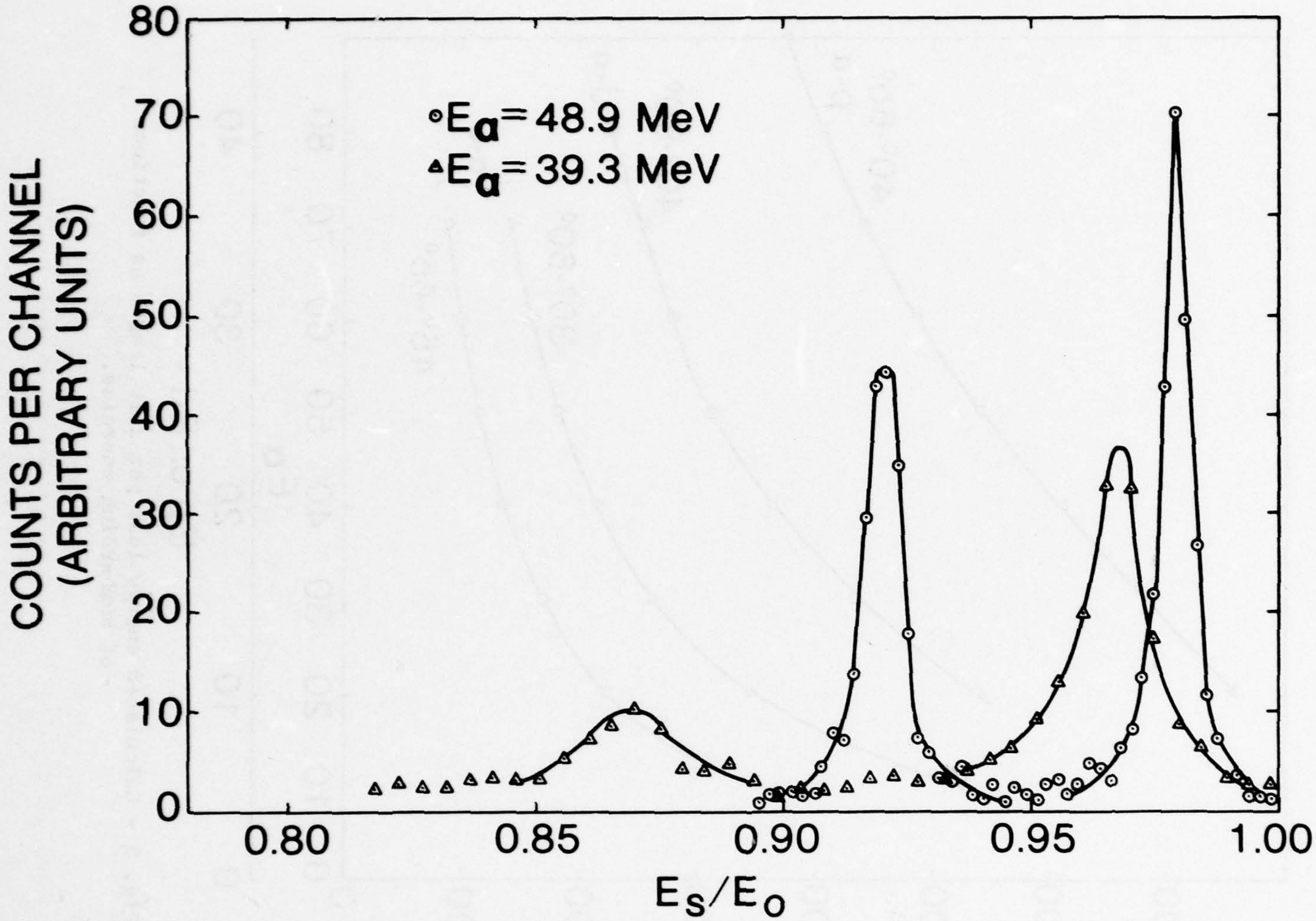

Fig. 4 - Similar to Fig. 3, except the undesirable energy for profiling represents another region with rapidly changing cross section to be avoided.

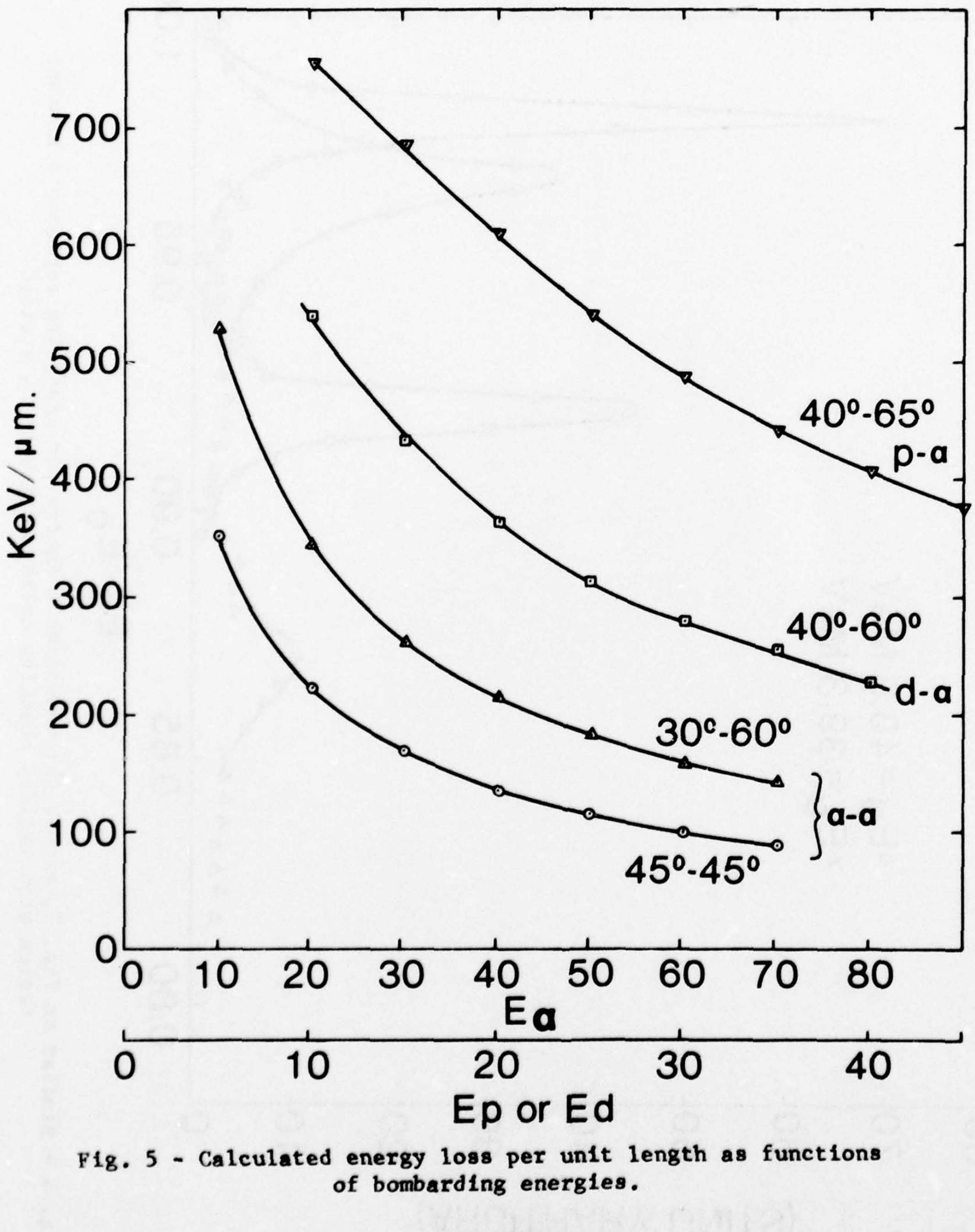

Fig. 5 - Calculated energy loss per unit length as functions of bombarding energies.

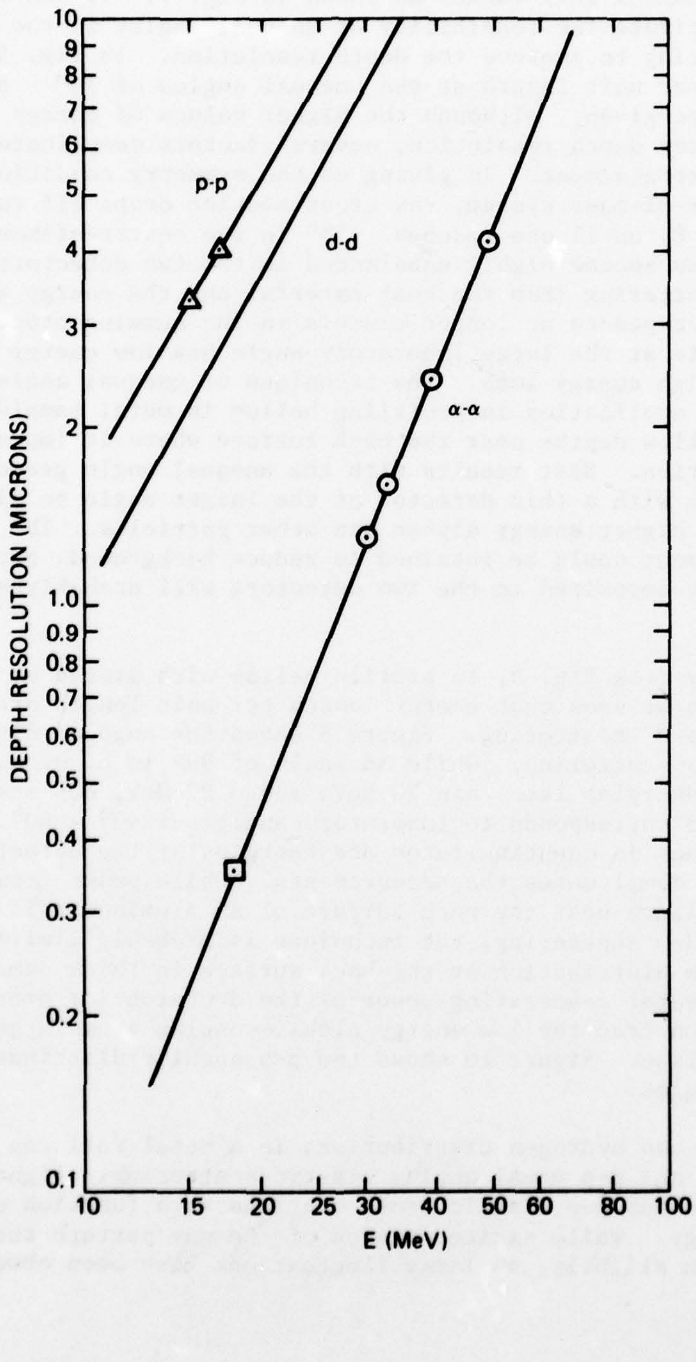

Fig. 6 - Depth resolutions for one-mil thick aluminum foils as function of bombarding energies for α-α, d-d, and p-p scattering.

followed so the incident energy falls in the same channel of the data system, the number of channels separating front and back implants on a one-mil aluminum foil varies as shown in Fig. 7. It was of interest to investigate the feasibility of unequal angles in the alpha-alpha scattering to improve the depth resolution. In Fig. 5 the energy loss per unit length at the unequal angles of 30^{o} - 60^{o} in the laboratory are given. Although the higher values of energy loss implies a greater depth resolution, several factors complicate this geometrical arrangement. In giving up the symmetry condition of 90^{o} in the center-of-mass system, the cross section drops off rapidly, as seen in Fig. 8, until one reaches 58^{o} in the center-of-mass. Total counting rates become highly unbalanced in the two detectors due to increased scattering from the host material and the energy spread due to angular acceptance no longer cancels in the summing process. The alpha particle at the large laboratory angle has low energy and, therefore, high energy loss. The technique of unequal angles would have limited application in profiling helium in metal samples, except for very shallow depths near the back surface where it improves the depth resolution. Best results with the unequal angle geometry would be obtainable with a thin detector at the larger angle to discriminate against higher energy alphas and other particles. The coincident requirement could be retained to reduce background, but summing of the energy deposited in the two detectors will probably not be useful.

Similarly from Fig. 5, to profile helium with proton or deuteron beams, it can be seen that energy losses per unit length are higher for d-α and p-α scattering. Figure 9 shows the angular distributions for d-α scattering. While an angle of 90^{o} in c. of m. is desirable for energies less than 20 MeV, above 23 MeV, 60^{o} seems more favorable and corresponds to laboratory angles of 40^{o} - 60^{o}. There is a difference in counting rates and energies of the detected particles which complicates the measurements. While peaks from delta function implants near the back surface of an aluminum foil have been observed by d-α scattering, the technique is probably limited to profiling helium distribution at the back surface in thick samples, using the greater penetrating power of the deuteron (or proton) and the resolution from the low energy alpha escaping at a large angle from the surface. Figure 10 shows the p-α angular distributions for several energies.

Deuteron and hydrogen distributions in a metal foil can be profiled by d-d and p-p equal angles elastic scattering. Figure 11 shows the d-d and p-p elastic cross sections as a function of bombarding energy. While excited states of ^{4}He may perturb the 90^{o} cross section slightly, no large fluctuations have been observed.

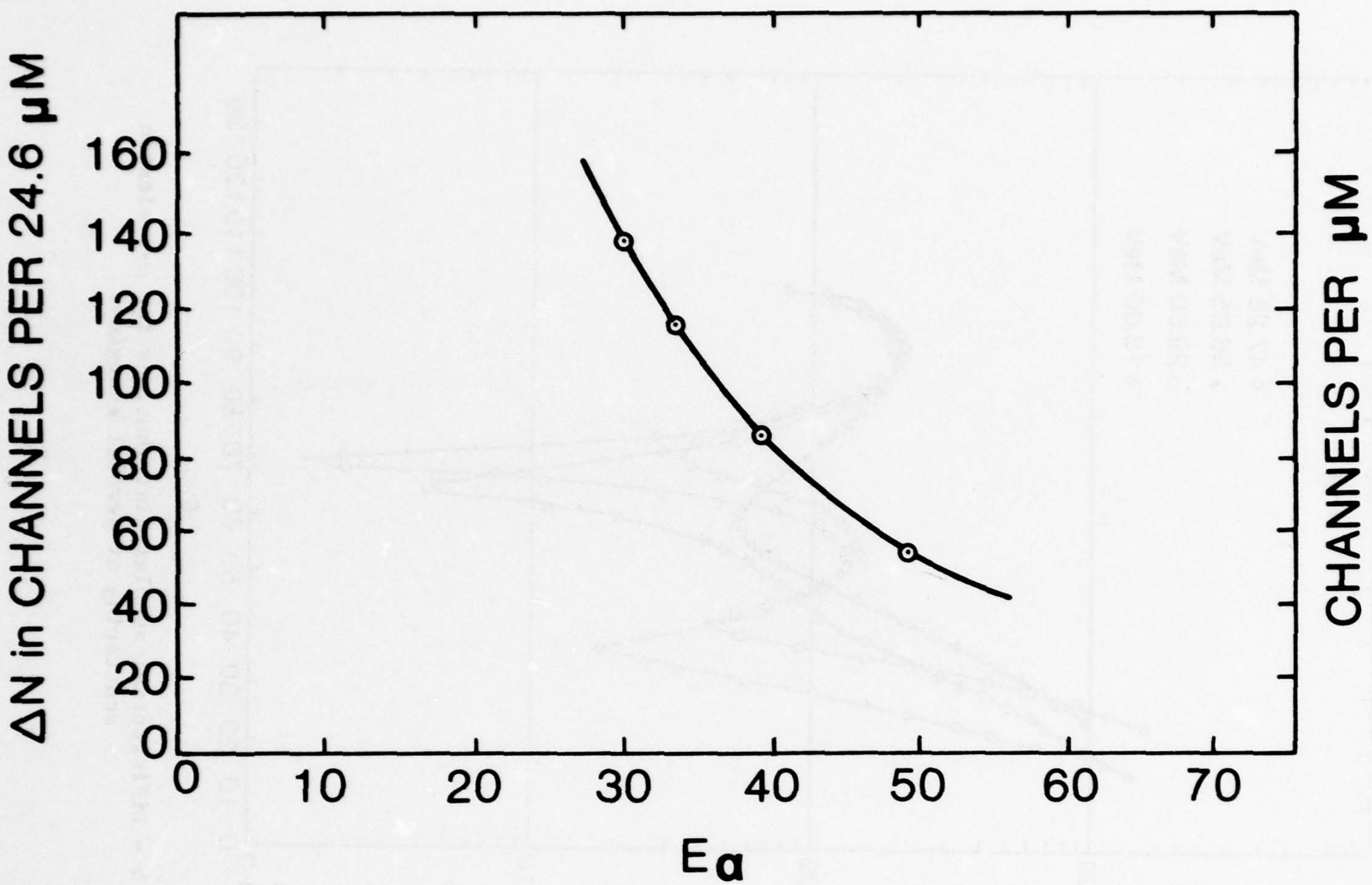

Fig. 7 - Number of channels separating front and back peaks as function of bombarding energy when amplifier gains are adjusted so the unscattered energy is in the same channel

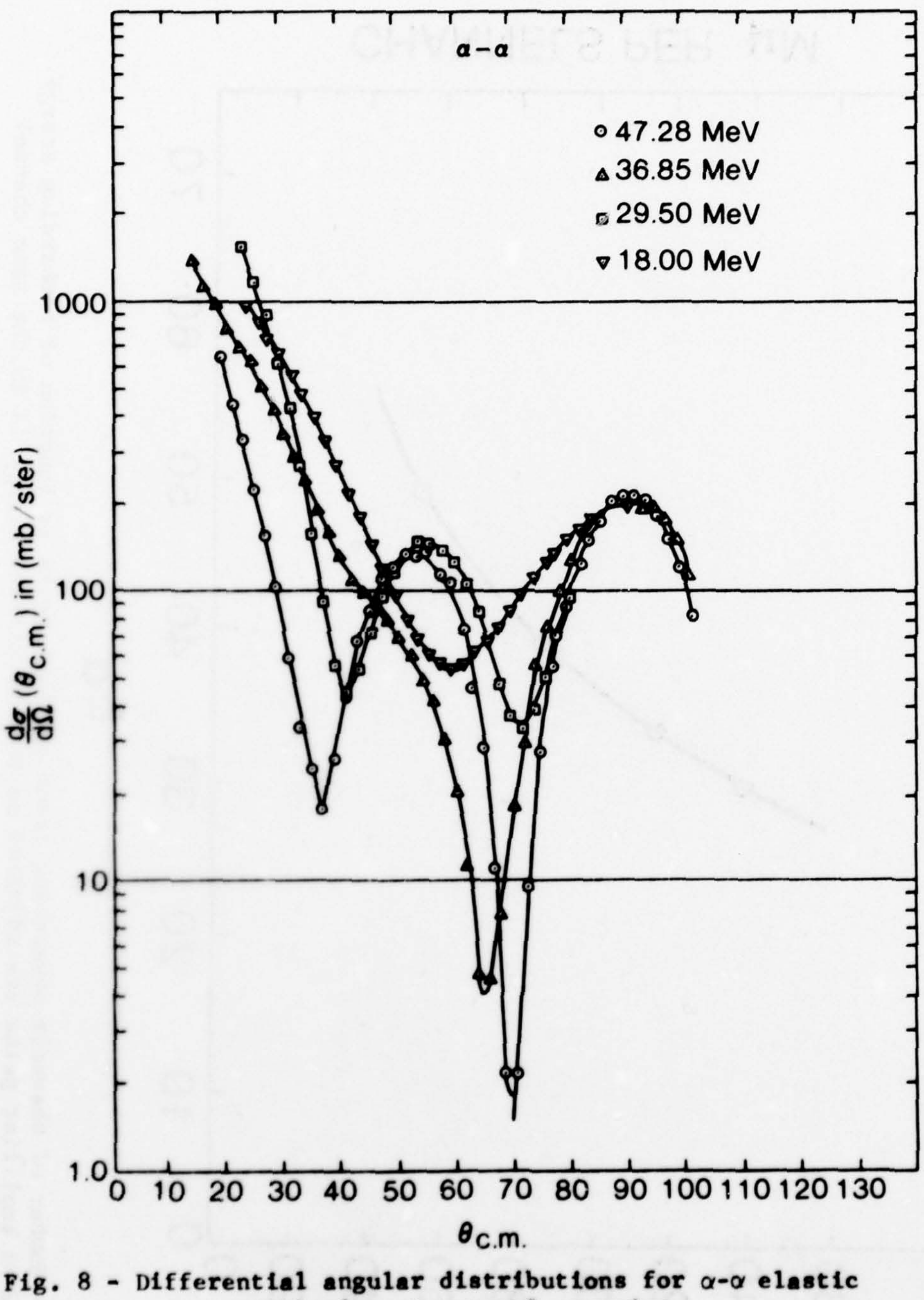

Fig. 8 - Differential angular distributions for α-α elastic scattering at several energies.

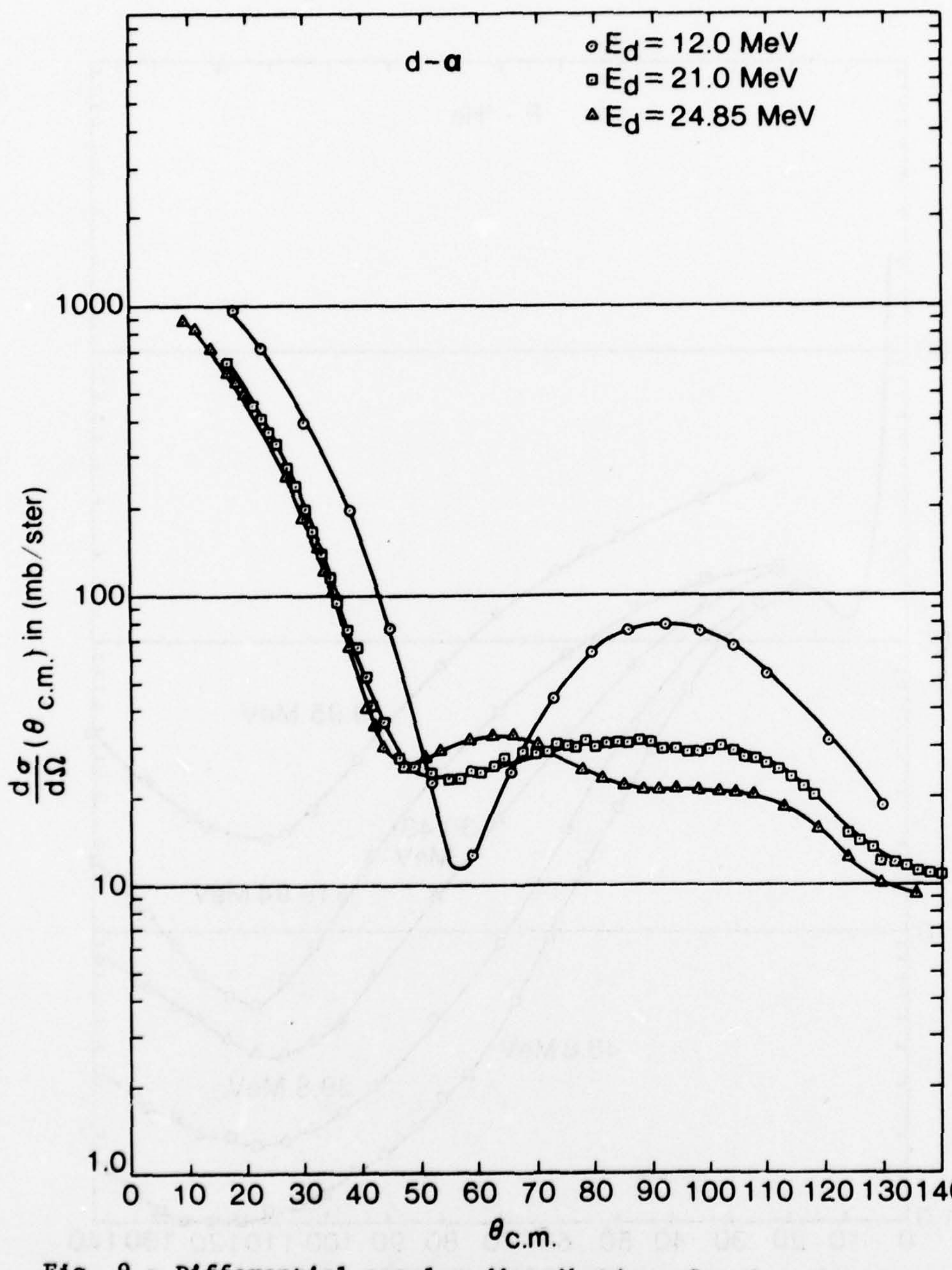

Fig. 9 - Differential angular distributions for d-α elastic scattering at several energies.

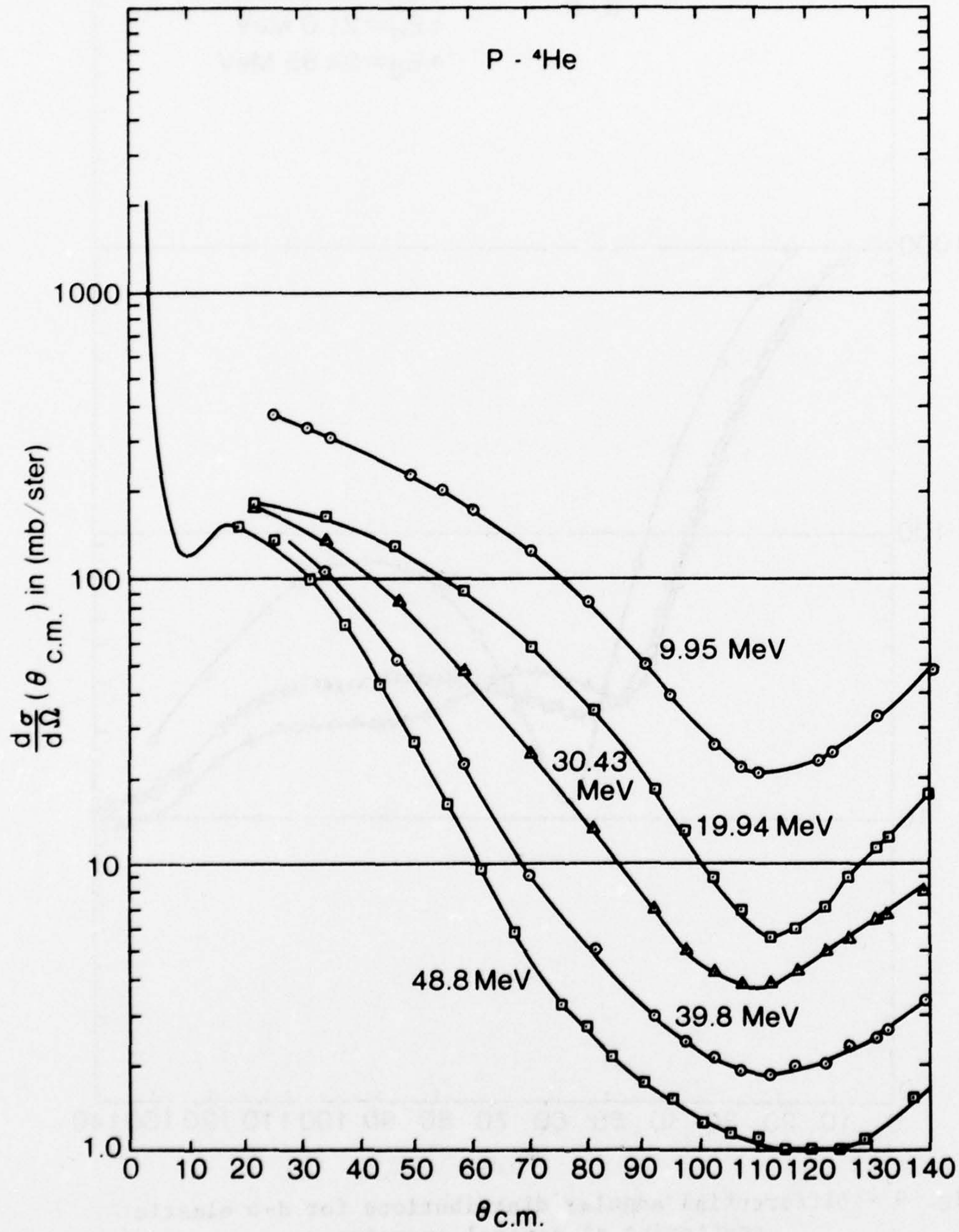

Fig. 10 - Differential angular distributions for p-α elastic scattering at several energies.

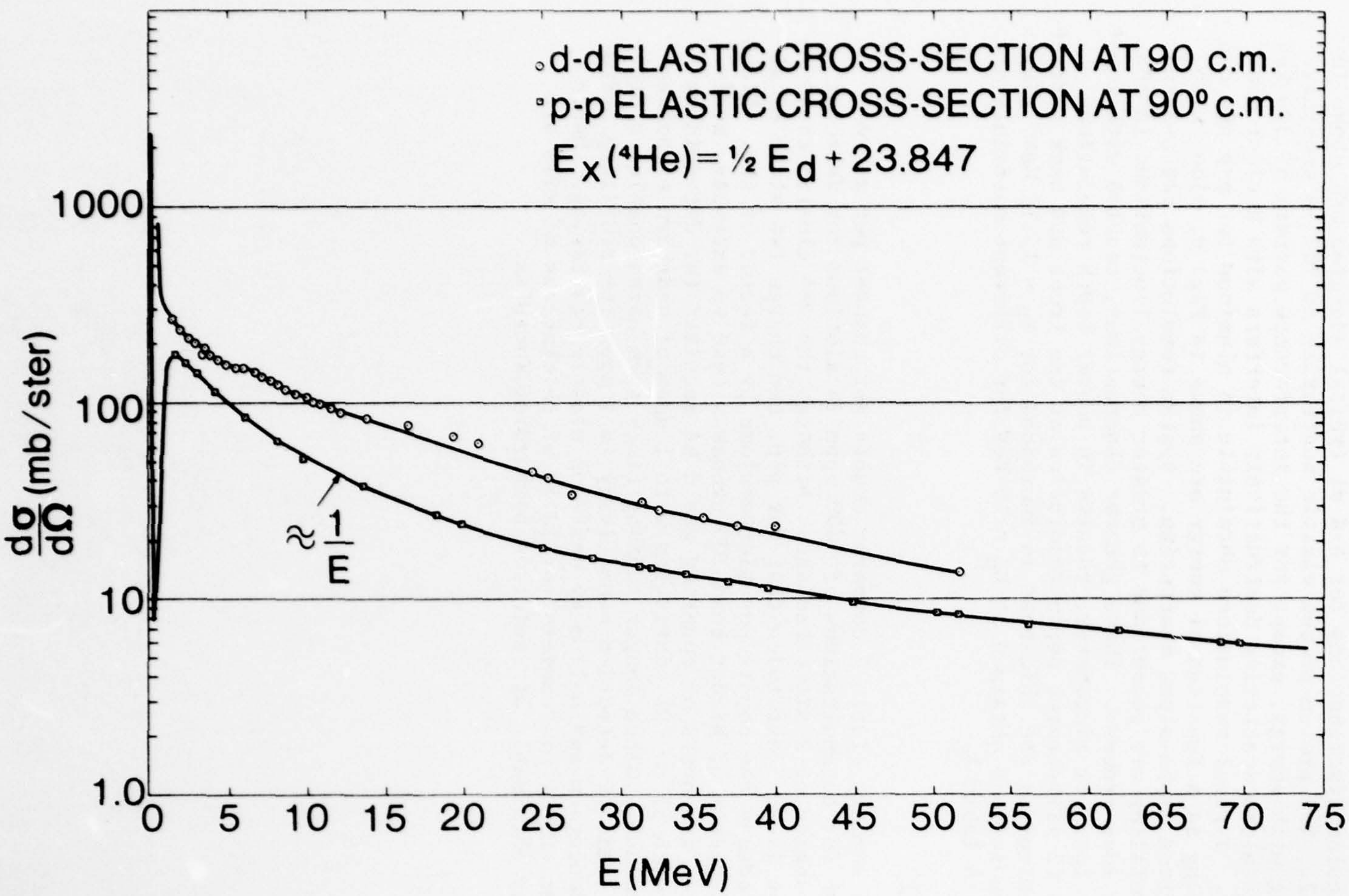

Fig. 11 - Elastic scattering cross sections at the center-of-mass angle of 90° for d-d and p-p. Neither curve shows any drastic variations with bombarding energy.

The angular distributions for d-d at typical energies are shown in Fig. 12. The proton-proton elastic scattering at 90^{o} is smoothly varying with energy, except for the interference pattern at 383 keV where coulomb scattering destructively interfers with nuclear scattering. Typical resolutions obtainable in aluminum for p-p and d-d profiling as a function of energy are shown in Fig. 6, along with those from alpha-alpha scattering. System resolutions for p-p and d-d profiling are poorer due to greater energy fluctuations in the energy loss process. These greater fluctuations, coupled with lower energy loss per micrometer, results in poorer depth resolution. Figure 13 illustrates depth resolution at the front and back surface of aluminum as the thickness is increased for E_p = 15.12 MeV. Typical resolutions obtained at E_p = 15 MeV for different materials are shown in Table 1.

<u>Summary</u>

In summary, Table 2 compares counts per channel per microcoulomb for concentrations of 1000 appm in aluminum for detector solid angles of 2 milsteradians. Although the α-α elastic cross section is at least twice that for p-p, the charge two state of alphas reduces the counts per microcoulomb by a factor of two. Since beam currents of higher than 100 nanoamps lead to excessive accidentals, ten hours of counting would be required for determining peaks with 5% to 10% statistics with 1 appm of hydrogen or helium in aluminum. Since longer running times seem unreasonable, a practical limit of detection sensitivity is 1 appm for profiling hydrogen, deuterium and helium by self-ion elastic scattering. Depth resolutions of micrometers for foils of thicknesses of mils are readily obtainable at cyclotron bombarding energies.

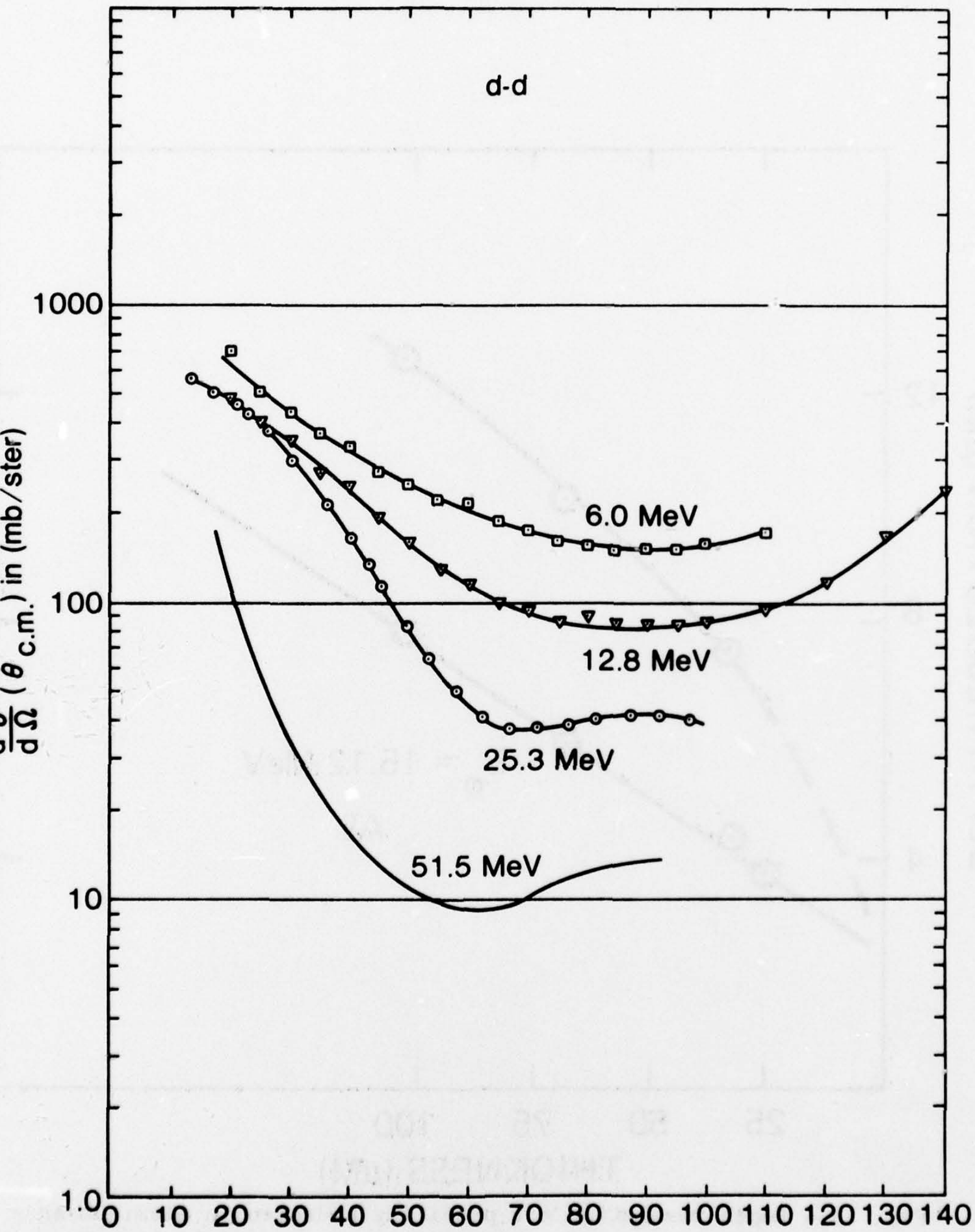

Fig. 12 - Differential angular distributions for d-d elastic scattering at several energies.

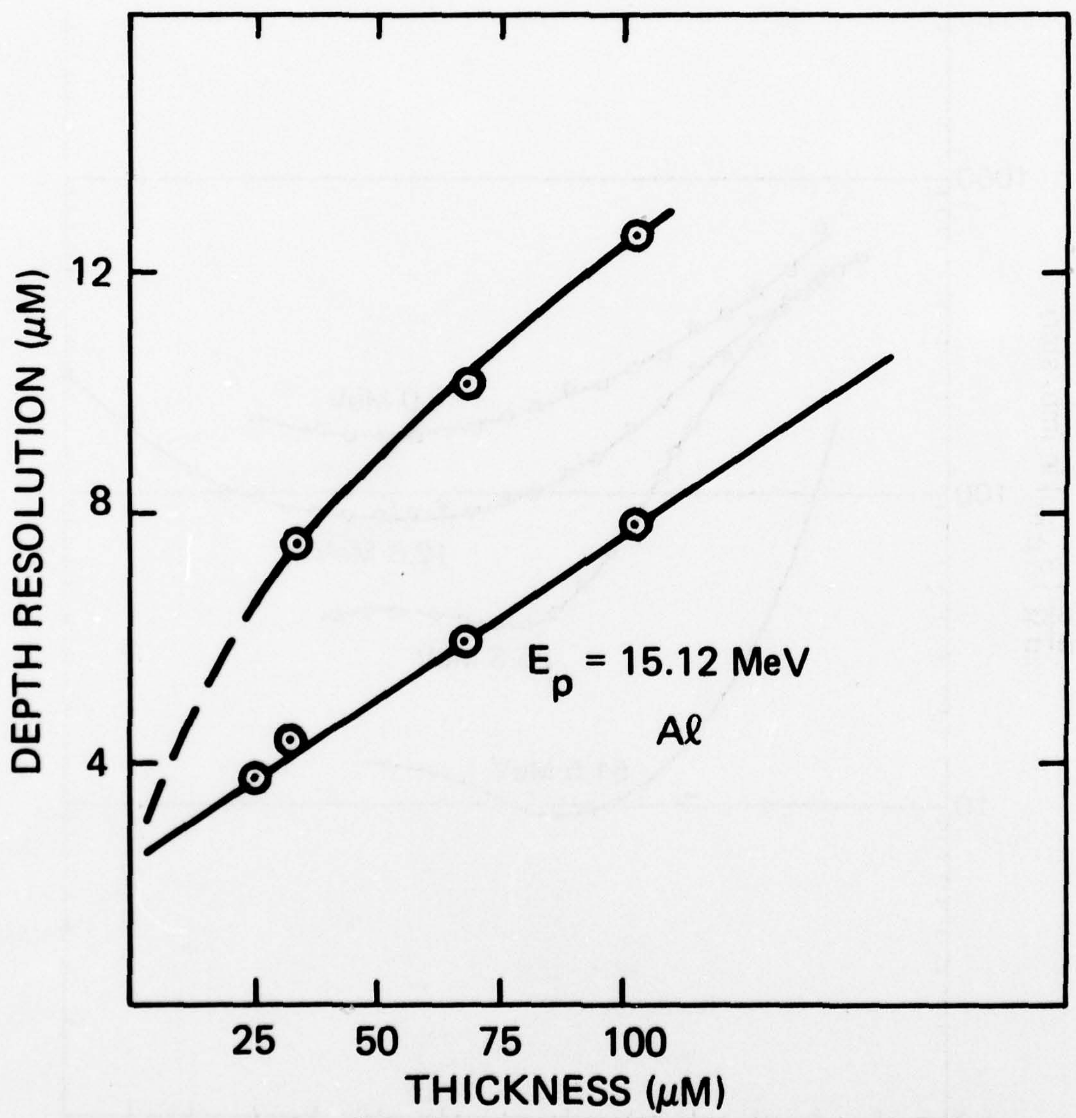

Fig. 13 - Depth resolution for profiling hydrogen in aluminum as a function of thickness for a bombarding energy of 15.12 MeV.

Table 1

Depth Resolutions for Various Materials for Profiling with $E_p = 15.12$ MeV.

Material	Foil Thickness (μM)	Resolution R(Back) (μM)	Resolution R(Front) (μM)	N Channels
Al	25	3.7	6.5	41
	34	4.4	7.5	50
	68	5.9	10	104
	102	7.5	12.6	163
Nb	33	3.4	5.0	106
SS	32	3.5	5.1	100
Ni	2	1.2	1.5	9

Table 2

Counts per channel per microcoulomb for 1000 appm of hydrogen, deuterium in aluminum with a two-milsteradian solid angle.

Energy (MeV)	$\frac{d\sigma}{d\Omega}$ c.m. (mb/ster)	keV/ch	N	Yield (ct/μc)
		p-p		
10	52	11.2	76	3.6
15	34	16.9	39	4.3
20	24	22.5	23	5.6
50	9	56.2	5	10.1
		d-d		
15	76	16.9	64	6.4
22	50	24.7	33	8.2
35	26	39.3	20	7.0
		α-α		
31	210	34.8	135	4.1
36.5	200	41.0	104	5.0
42.0	250	47.1	80	8.2
47.5	210	53.4	59	9.3

B. Transient Irradiation-Induced Creep of Nickel During Deuteron Bombardment

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Background

Experiments to investigate irradiation-induced creep of materials using ion bombardment techniques have been successfully conducted by various investigators (7-11). The results obtained from these experiments have provided valuable insight concerning the phenomenon of irradiation creep as well as providing results for comparison with reactor data and with theory. In most cases, the experiments have been concerned with the investigation of irradiation creep under steady-state conditions. However, some experiments have shown that large transient creep strains can occur in nickel during irradiation periods as short as 6 to 12 hours (7).

In the previous CORES report (12), experimental results were presented from an investigation of the transient creep behavior of cold worked nickel at 224°C for periods up to eight hours. Preliminary results from a quantitative characterization of the transient creep microstructure also were presented. During the current reporting period, the microstructural characterization was completed and these results and conclusions reached are presented in this report.

Progress

A complete description of the experimental procedures used for the creep experiments and microscopy studies of the irradiated specimens has been given in previous reports (9,13). The quantitative microstructural results from the creep specimens are summarized in Table 3, along with the previously reported results for specimen 4D-4 and for unirradiated, unstressed material (13). As discussed in the previous CORES report (12), the results in Table 3 show that the dislocation density, defect cluster/loop number density, and mean defect cluster/loop diameter after eight hours of irradiation have achieved values which are nearly equal to those obtained for the specimen irradiated for 23 hours. However, the specimen irradiated for only four hours exhibited a nearly 50% higher dislocation density with slightly lower defect cluster/loop number density and smaller mean defect cluster/loop diameter. After only two hours irradiation, the

Table 3

Summary of Creep Rates and Microstructural Results for Nickel

Specimen Number	Irradiation Test Temperature, °C	Stress MPa	Irradiation Time Hrs.	Displacement rate x 10^8 dpa/s	Fluence x 10^2, dpa	Defect Cluster/Loop Number Density, #/cm^3	Mean Defect Cluster Loop Dia., Å	Dislocation Density, cm/cm^3	Measured Creep Rate x 10^2, dpa^{-1}	Calculated Climb-Glide Creep Rate x 10^2, dpa^{-1}
4D-4-2	224	345	23	27.17	2.3	1.4×10^{16}	43	3.2×10^{10}	4.9	4.7
6-29	224	345	8	27.17	0.8	1.3×10^{16}	39	3.3×10^{10}	7.8	5.4
6-28	224	345	4	27.17	0.4	1.1×10^{16}	34	4.8×10^{10}	9.0	9.5
6-34	224	345	2	27.17	0.2	8.2×10^{15}	29	6.6×10^{10}	14.3	16.7
4D	Unirradiated, unstressed material					$< 10^{13}$	30	$> 2 \times 10^{11}$	0.01	440.0

dislocation density was twice the value at eight hours with a 40% lower defect cluster/loop number density. The variation of the defect cluster/loop number density and dislocation density as a function of irradiation time is shown in Fig. 14.

Previous work employing calculations, based on the microstructural data, has confirmed that a mechanism of climb-controlled glide of dislocations was in reasonable agreement with the experimentally observed creep behavior of nickel specimens irradiated from 15 to 25 hours at 224°C (13). In the present study, however, interpretation of the transient creep behavior in terms of the microstructure requires the identification of the components which appear to be stabilized at irradiation times of approximately eight hours. Figure 14 suggests that, since both the defect cluster/loop number density and the network dislocation density satisfy this criterion, both components were important during transient creep. However, since it is the motion of individual dislocations which provide the primary contribution to the creep strain, the network dislocation density can be considered to be the microstructural component which primarily controls the transient creep stage. As suggested by neutron irradiation results (14), these dislocations may migrate by irradiation-induced climb which permits them to move to points of annihilation. Furthermore, under the influence of the applied stress, directed dislocation glide may occur in conjunction with the irradiation-induced climb to produce the creep strain. Direct evidence that both climb and glide dislocation motion occurs during irradiation creep has been obtained from high voltage electron microscopy studies (15). Nevertheless, the rapidly increasing number density of defect clusters/loops will provide important obstacles to the dislocation motion such that a climb-controlled glide process may be the controlling mechanism during the transient creep stage as well as during steady-state creep.

In order to test whether the climb-controlled glide mechanism confirmed for the steady-state results could be applicable to the present study, the agreement between the experimental transient creep rates was compared with transient creep rates calculated on the basis of the microstructural data. For purposes of the comparison, the expressions previously employed for the steady-state calculations (13), were used to obtain approximate values of the transient creep rates for the specimens irradiated for two, four and eight hours from the results in Table 3. A hypothetical creep rate was calculated at time $t \approx 0$ from the microstructural data for the unirradiated, unstressed specimen to obtain approximate values at the instant the ion beam struck the specimens. The comparison of the calculated and experimental creep rates is shown in Fig. 15. It is evident from Fig. 15 that the transient creep rates calculated using a climb-controlled

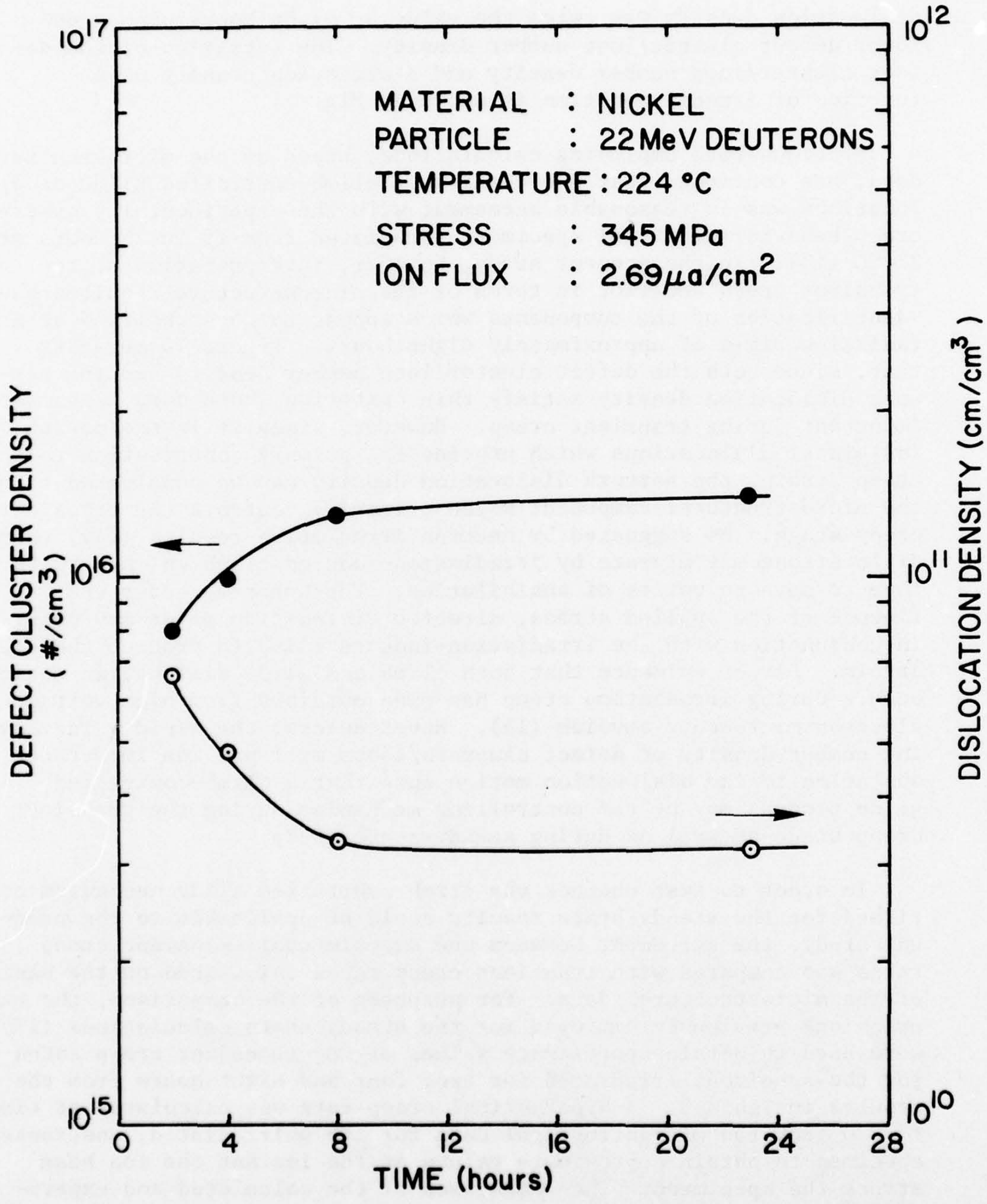

Fig. 14 - Variation of measured defect cluster/loop number density and dislocation density as a function of irradiation time.

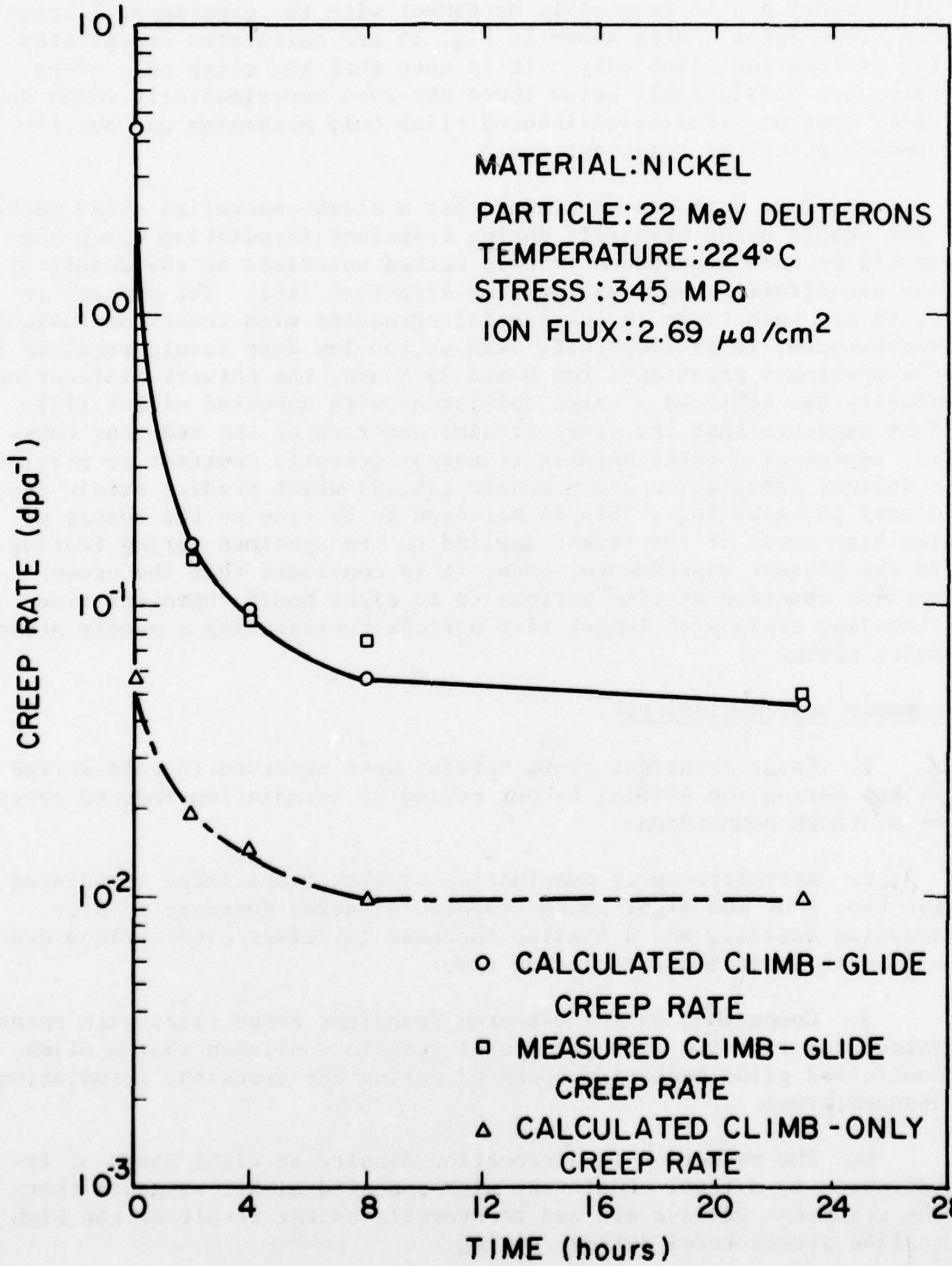

Fig. 15 - Comparison of the calculated and experimental creep rates.

glide model are in reasonable agreement with the experimental transient creep rates. Also shown in Fig. 15 are calculated creep rates for dislocation climb only. It is seen that the climb only creep rates are considerably below those observed experimentally which suggests that an irradiation-induced climb only mechanism was not responsible for the transient creep.

Previous work has concluded that a climb-controlled glide mechanism should occur primarily during transient irradiation creep and should be more pronounced in cold worked materials as the result of the nonuniform network dislocation structure (16). The present results are seen to be in substantial agreement with these conclusions. Furthermore, it is seen that, even at the low dose levels received by the specimens irradiated for 8 and 23 hours, the network dislocation density has achieved a value consistent with annealed nickel (17). This suggests that the creep strains observed at two and four hours may represent non-recoverable transient creep in contrast to previous transient irradiation creep models (18,19) which predict strain recovery on unloading. This is believed to be true as the result of the high level of the stress applied to the specimen during testing. In the present experiments, then, it is concluded that the creep strains observed at time periods up to eight hours represent true transient creep with longer time periods representing a nearly steady-state creep.

Summary and Conclusions

1. Large transient creep strains were observed in cold-worked nickel during the initial 8-hour period of irradiation-induced creep by deuteron bombardment.

2. Microstructural examination of nickel specimens irradiated for two, four and eight hours revealed a marked decrease in dislocation density, and a similar increase in defect cluster/loop density with increased irradiation time.

3. Comparison of the measured transient creep rates with rates calculated from the microstructural results confirmed that a climb-controlled glide mechanism operated during the transient irradiation-induced creep.

4. The reduction in dislocation density at eight hours of irradiation to a level consistent with annealed nickel suggests that the transient strains are non-recoverable as the result of the high applied stress level during testing.

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